

## PATENT ABSTRACTS OF JAPAN

(11)Publication number : 2001-261375

(43)Date of publication of application : 26.09.2001

(51)Int.Cl.

C03C 17/22  
// C23C 16/34  
C23C 16/42

(21)Application number : 2000-070477

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(22)Date of filing : 14.03.2000

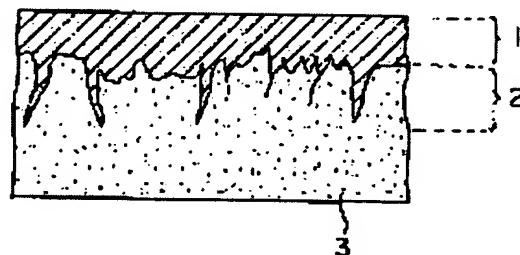
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## (54) CERAMIC-COATED QUARTZ GLASS BODY

(57)Abstract:

**PROBLEM TO BE SOLVED:** To obtain a ceramic-coated quartz glass body having neither peeling nor damage of a ceramic layer even if repeatedly subjected to thermal shocks being controlled in dust, etc., having excellent chemical resistance and excellent handleability.

**SOLUTION:** This ceramic-coated quartz glass body is obtained by coating the surface of a porous quartz glass body 3 or the surface a porous quartz glass layer formed on a quartz glass body with ceramic layers 1 and 2. The ceramic-coated quartz glass body is produced by forming a ceramic layer on the surface of the porous quartz glass body or the porous quartz glass layer by a chemical vapor-phase growth reaction, then taking out the porous quartz glass from a furnace and cleaning the ceramic layer with an acid.



## LEGAL STATUS

[Date of request for examination]

03.03.2003

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's decision of

rejection]

[Date of requesting appeal against examiner's  
decision of rejection]

[Date of extinction of right]

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CLAIMS

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[Claim(s)]

[Claim 1] The ceramic covering quartz-glass object characterized by covering with a ceramic layer the front face of a porosity quartz-glass object, or the front face of a porosity quartz-glass layer established in the quartz-glass object.

[Claim 2] The ceramic covering quartz-glass object according to claim 1 characterized by said ceramic layer consisting of silicon carbide or silicon nitride.

[Claim 3] The ceramic covering quartz-glass object according to claim 1 or 2 characterized by being the ceramic layer in which said ceramic layer was formed by the chemical-vapor-deposition method.

[Claim 4] The ceramic covering quartz-glass object according to claim 1 to 3 characterized by consisting of a surface part of gas impermeability which said ceramic layer becomes from ceramic singleness substantially, and a inner layer part which has the ceramic infiltration organization in which the ceramics permeated in the detailed pore of a porosity quartz-glass object or a porosity quartz-glass layer.

[Claim 5] The ceramic covering quartz-glass object according to claim 4 which is in the range whose thickness of the surface part of said ceramic layer is 10 thru/or 1000 micrometers, and is characterized by the thickness of said inner layer part being 500 micrometers or more.

[Claim 6] The ceramic covering quartz-glass object according to claim 1 to 5 characterized by the thickness of the porosity quartz-glass layer prepared in said quartz-glass body surface being 1 thru/or 10mm.

[Claim 7] The consistency of said porosity quartz-glass object or the consistency of the porosity quartz-glass layer prepared in the quartz-glass body surface is 0.1 thru/or 1.95g/cm<sup>3</sup>. Ceramic covering quartz-glass object according to claim 1 to 6 characterized by being in the range.

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## DETAILED DESCRIPTION

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### [Detailed Description of the Invention]

[0001]

[Field of the Invention] This invention relates to tough and the ceramic covering quartz-glass objects in which the ceramic layer excellent in corrosion resistance and abrasion resistance was formed, such as silicon carbide and silicon nitride, about a ceramic covering quartz-glass object on the front face of a porosity quartz-glass layer established in the front face of the porosity quartz-glass object which reinforcement \*\*\*\* in a detail easily weakly and carries out raising dust to it more, or the quartz-glass body surface.

[0002]

[Description of the Prior Art] In the semi-conductor manufacture process, various kinds of members which consist of a porosity quartz-glass object or a quartz-glass object are used widely. For example, porosity quartz-glass objects, such as foaming quartz glass (nature foam of silica glass), are widely used in the various furnaces for semi-conductor processing as structure heat insulators, such as a heat insulating board and a wallplate, functional heat insulators, such as a wafer boat table, etc. Moreover, the quartz-glass object is widely used for the tubular Plastic solid of tabular Plastic solids, such as a thermal shield plate, a heat-resistant nozzle, a coil, etc., etc., and the wafer boat regardless of the transparent body and the opaque body.

[0003] Said porosity quartz-glass object is lightweight compared with a quartz-glass object, thermal conductivity is low, and it has the property which was excellent as a heat insulator. However, it being difficult to use a general-purpose article as it is in the semi-conductor manufacture process field which dislikes contamination according to dust in order to \*\*\*\* the usual porosity quartz-glass object easily and to carry out raising dust to the degree of pole, and quartz glass's usually enclosing a porosity quartz-glass object, etc. and a certain raising dust control measures are taken, and it is used as after, a heat insulator, etc.

[0004]

[Problem(s) to be Solved by the Invention] By the way, the quartz-glass member which enclosed the porosity quartz-glass object, or the member for semi-conductor manufacture processes which consists of a quartz-glass object is washed using acid cleaning liquid, such as a fluoric acid water solution, in case it is used for defecation. Therefore, when it was used to some extent, by said washing, etching might progress and shakiness and breakage might take place between members. In order to solve this problem (i.e., in order to raise the chemical resistance (chemical resistance) of the member which consists of a quartz-glass object), tough and the researches and developments covered with the ceramic layer excellent in corrosion resistance and abrasion resistance of silicon carbide (SiC), silicon nitride (Si<sub>3</sub>N<sub>4</sub>), etc. are furthered in the front face.

[0005] however, when covering formation of the ceramic layers, such as the direct above-mentioned silicon carbide and silicon nitride, is carried out on the front face of a quartz-glass object Since there is a difference of the coefficient of thermal expansion ( $5.6 \times 10^{-7}$ /degree C) of a quartz-glass object and the coefficient of thermal expansion ( $4 \times 10^{-6}$ /degree C) of ceramics, such as said silicon carbide, When the heat histories, such as repeat heating and cooling, were received, the film (layer) destruction and exfoliation resulting from this thermal shock distortion might arise, and it might become the cause of dust.

[0006] The approach of controlling generating of such dust etc. is also proposed. To JP,8-83835,A In the quartz-glass base material which carried out surface coating with silicon carbide between this quartz-glass base material front face and a silicon carbide membrane layer For example, like silicon and silicon nitride, the inclination layer of the addition component which consists of the matter also with small degree of hardness and elastic modulus rather than silicon carbide is made to intervene as an interlayer, and the silicon carbide covering quartz-glass object which buffered heat distortion generating which originates in the difference of said thermal

expansion by this interlayer is proposed. The silicon carbide covering quartz-glass object of the above-mentioned structure has good thermal shock resistance, and has the resistance stabilized in the long run about the chemical resistance force over a fluoric acid water solution, nitric-acid water solutions, these mixed-acid water solutions, etc., and dust raising dust control and diffusion evasion of semi-conductor harmful matter.

[0007] However, when the 3rd component other than a quartz-glass object and a silicon carbide component, especially a silicon ingredient were included in this way, and there was an increment in weight at this rate and it used as various members of the member for semi-conductor manufacture processes, the difficulty was in handling nature. Moreover, in order that heat capacity might increase-ize, when it used as a heat treatment member, there was a trouble that heat responsibility was not enough etc.

[0008] This invention aims at offering the ceramic covering quartz-glass object which does not have exfoliation or breakage of a ceramic layer even if it receives the thermal shock of a repeat, and raising dust, such as dust, was controlled, and was moreover excellent in chemical resistance (chemical resistance), and was excellent in handling nature.

[0009]

[Means for Solving the Problem] The ceramic covering quartz-glass object concerning this invention made in order to solve the above-mentioned technical technical problem is characterized by covering with a ceramic layer the front face of a porosity quartz-glass object, or the front face of a porosity quartz-glass layer established in the quartz-glass object. It is desirable for said ceramic layer to consist of silicon carbide or silicon nitride here, and it is desirable that it is the ceramic layer in which said ceramic layer was formed by the chemical-vapor-deposition method.

[0010] Moreover, it is desirable to consist of a surface part of gas impermeability which said ceramic layer becomes from ceramic singleness substantially, and a inner layer part which has the ceramic infiltration organization in which the ceramics permeated in the detailed pore of a porosity quartz-glass object or a porosity quartz-glass layer, it is in the range whose thickness of the surface part of said ceramic layer is 10 thru/or 1000 micrometers, and it is desirable for the thickness of said inner layer part to be 500 micrometers or more.

[0011] Furthermore, the consistency of the porosity quartz-glass layer which it was desirable for the thickness of the porosity quartz-glass layer prepared in said quartz-glass body surface to be 1 thru/or 10mm, and was prepared in the consistency or quartz-glass body surface of said porosity quartz-glass object is 0.1 thru/or 1.95 g/cm<sup>3</sup>. It is desirable that it is in the range.

[0012] The ceramic covering quartz-glass object of this invention has the description on a configuration in tough and the points with which it is the ceramic layer excellent in corrosion resistance and abrasion resistance, and the front face of a porosity quartz-glass object or the front face of a porosity quartz-glass layer established in the quartz-glass object is covered, such as silicon carbide and carbonization nitrogen.

[0013] When ceramic layers, such as silicon carbide (SiC), were covered on the front face of the usual quartz-glass object which is not a porous body, as already explained, it originated in the difference of the coefficient of thermal expansion of a quartz-glass object and a ceramic layer, a crack and membrane layer exfoliation arose in the ceramic layer, and this had become causes, such as dust, on it. On the other hand, with the ceramic covering quartz-glass object of this invention, ceramic covering of SiC etc. is given to the front face of a porosity quartz-glass object, or the front face of a porosity quartz-glass layer established in the quartz-glass object, namely, a ceramic layer is formed in it.

[0014] Unlike the case of the above mentioned quartz-glass object, the ceramic layer prepared in the front face of a porosity quartz-glass layer established in the front face of a porosity quartz-glass object or the quartz-glass object permeates from an interface in the detailed pore inside a porosity quartz-glass object or a porosity quartz-glass layer. That is, while the surface part of gas impermeability which consists of ceramic singleness, such as SiC, substantially is formed on said interface, the inner layer part of the infiltration organization in which said ceramics permeated in the detailed pore of a porous body is formed in the bottom of this interface. By existence of this inner layer part, the exfoliation and breakage of the ceramic layer used as a surface accompanying the coefficient-of-thermal-expansion difference of a quartz-glass object and ceramics, such as SiC, can be prevented.

[0015] If it is in the porosity quartz-glass layer prepared in the porosity quartz-glass object of this invention, and the quartz-glass object, the quartz glass is divided by countless detailed pore, and exists as organization which the very thin septum side which divides these followed. Therefore, since it is flexible and the detailed septum of quartz glass moreover has the complicated configuration by the area of jointing with ceramic layers, such as

SiC, becoming large compared with the quartz-glass object to the flow stress by a thermal shock etc., respectively as compared with the quartz-glass object, stress distribution becomes easier. direct on a quartz-glass object, even if it receives the heat history like repeat heating and cooling for a long period of time, when ceramic layers, such as SiC, are covered also with this to said porous body which was carried out -- neither film (layer) exfoliation nor the breakage by the interface occurs like [ at the time of covering SiC etc. ], but it becomes what was extremely excellent in raising dust checked.

[0016] Moreover, in the case of a SiC layer etc., the ceramic layer of corrosion resistance is high, therefore possible also for carrying out acid cleaning of the whole member. Moreover, the effectiveness that the reinforcement of the member itself increases is also acquired by covering with tough ceramics, such as SiC.

[0017] Since especially the ceramic covering quartz-glass object that consists of the porosity quartz-glass object which covered the ceramic layer of this invention uses the porosity quartz-glass object for the base material, in addition to excelling in many effectiveness, such as the above-mentioned dust raising dust checked, impurity diffusion checked, corrosion resistance, and tough nature, it is lightweight, and is high, and does so the effectiveness which was extremely excellent as a heat insulation member for semi-conductor manufacture processes. [ of adiabatic efficiency ]

[0018] Moreover, the ceramic covering quartz-glass object which consists of the porosity quartz-glass object which covered the ceramic layer of this invention can obtain the layered product which has good adhesion easily by welding a porosity quartz-glass object to the front face of a quartz-glass object. Moreover, with the ceramic covering quartz-glass object concerning this invention which covered with the ceramic layer the front face of a porosity quartz-glass layer established especially in the quartz-glass object, a porosity quartz-glass object serves as a buffer coat, and ceramics, such as SiC, becomes thermal shock resistance from the interface of this and a porosity quartz-glass object by entering into internal pore with what was extremely excellent. And since the adhesion of a porosity quartz-glass layer and ceramic layers, such as SiC, is also good, it excels in raising dust checked, such as dust, and, moreover, a ceramic layer becomes what in the case of the high grade SiC membrane layer formed by the CVD forming-membranes method etc. was excellent also in impurity diffusion checked and was extremely excellent as a member for semi-conductor manufacture processes, as already stated.

[0019] In addition, the above-mentioned ceramic covering quartz-glass object of this invention Process the configuration of a request of a porosity quartz-glass object, set the acquired porosity quartz-glass object on the support in a furnace, and the ceramic layer of gas impermeability is made to form in this porosity quartz-glass body surface by the chemical-vapor-deposition reaction. Subsequently, after \*\*\*\*(ing) and carrying out acid cleaning, it sets at the time of the front ceramic stratification. It is desirable to support a different part from the part which was in contact with said support of said porosity quartz-glass object with the support in a furnace, and to manufacture by forming the surface part of the ceramic layer of request thickness by the chemical-vapor-deposition reaction further.

[0020] For example, if a porosity quartz-glass object tends to be set and it is going to form the surface part of the ceramic layer of desired thickness (for example, 500-1000 micrometers) with 1 time of chemical vapor deposition on the support supported three points In case it will be in the condition that the suitable ceramic film was formed also in the part which touches the support of a porosity quartz-glass object, and support was joined to the porosity quartz-glass object and a porosity quartz-glass object is \*\*\*\*(ed), the fault which this vitreous humour itself damages will arise. Moreover, although it does not damage, the ceramic film (layer) will not fully be formed in the part which was in contact with said support, but gas impermeability will become inadequate.

[0021] Moreover, when a base material is a quartz-glass object, a porosity quartz-glass layer is joined to the front face of the quartz-glass object into which the desired configuration was processed by welding. Form, set said acquired quartz-glass object on the support in a furnace, and a chemical-vapor-deposition reaction is made to perform. In the time of the front [ after making the ceramic layer of gas impermeability form in this porosity quartz-glass layer front face, \*\*\*\*(ing) subsequently and carrying out acid cleaning ] ceramic stratification It is desirable from the same reason as the above to support a different part from the part which was in contact with said support of said quartz-glass object with the support in a furnace, and to manufacture by forming the surface part of the ceramic layer of request thickness by the chemical-vapor-deposition reaction further.

[0022]

[Embodiment of the Invention] This invention is explained more below at a detail. The ceramic covering quartz-glass object of this invention is the description on a configuration of the point that the front face of a

porosity quartz-glass object or the front face of a porosity quartz-glass layer established in the quartz-glass object is covered with the ceramic layer. It is possible for a porosity quartz-glass object to use a material equivalent to usual quartz glass as a raw material, for it to be processed and obtained and for thermal resistance, chemical stability, purity, etc. to lightweight-ize apparent density for this to about about 1 of usual quartz glass / 10 generally, according to quartz glass porosity-ization. And said porosity quartz-glass object is excellent also in those with low-thermal expansibility, and workability with high thermal resistance. The nature porous body of a silica (apparent density: 1.00 - 1.99 g/cm<sup>3</sup>, preferably 1.80 - 1.95 g/cm<sup>3</sup>) and the nature foam of silica glass (apparent density: 0.1 - 0.99 g/cm<sup>3</sup>, preferably 0.1 - 0.50 g/cm<sup>3</sup>) which indicate below the porosity quartz-glass object which can be used by this invention are included.

[0023] Namely, are shown in JP,4-202070,A as an example of such a porosity quartz-glass object. Add the amorphous silica powder of a very detailed particle size to crystalline substance silica powder, and it mixes to it. The nature porous body of a silica which is calcinated [ porous body ] after shaping, was obtained [ porous body ], carried out the partial bond of this crystal silica \*\*\*\* for the crystalline substance silica grain front face densely by the wrap amorphous silica impalpable powder layer, and made many open pores form between crystal silica \*\*\*\*, Moreover, contain the hydroxyl group shown in JP,5-345636,A, and specific surface area heat-treats the amorphous silica base material 6m<sup>2</sup> / more than g in the ambient atmosphere containing 600 thru/or 1300-degree C ammonia gas. Subsequently, the nature foam of high grade silica glass which was made to carry out heating foaming and was obtained in the temperature requirement (1350 degrees C thru/or 1800 degrees C), Furthermore, specified quantity addition of the high grade silicon nitride by which particle-size control was carried out at the nature raw material powder of high grade silicic acid shown in JP,5-254882,A is carried out, and the bubble diameter distribution 20 thru/or 180-micrometer nature foam of silica glass, etc. obtained by fusing by the oxyhydrogen flame is mentioned. It sets to this invention also especially among these porosity quartz-glass objects, and they are a consistency 0.1 thru/or 1.95 g/cm<sup>3</sup>. It is desirable from a viewpoint of contamination tightness, lightweight nature, adiathermic, and heat-capacity balance to use a high grade article.

[0024] In this invention, surface coating of the front face of the above-mentioned porosity quartz-glass object is carried out by tough and the ceramic film (layer) excellent in corrosion resistance and abrasion resistance, such as silicon carbide (SiC) and silicon nitride (Si<sub>3</sub>N<sub>4</sub>). SiC and Si<sub>3</sub>N<sub>4</sub> which are formed in the front face of this porosity quartz-glass object etc. -- as shown in drawing 1 as a mimetic diagram, as for the film (layer), it is desirable to consist of the surface part 1 of gas impermeability which membranous cross-section organization becomes from ceramic singleness substantially, and the inner layer part 2 of the infiltration organization in which the ceramics permeated in the detailed pore of the porosity quartz-glass object 3.

[0025] And if it is in the ceramic covering quartz-glass object which carried out SiC covering of the porosity quartz-glass object again, as for the thickness of the gas impermeable surface section 1 of said SiC singleness, it is desirable from a chemical-resistant and dust raising dust checked point that it is in 10 thru/or the range of 1000 micrometers. Moreover, as for the thickness of the inner layer part 2 which has the SiC infiltration organization in which SiC permeated in the detailed pore of said porosity quartz-glass object, it is desirable that it is 3mm or more from the viewpoint which collateralizes reinforcement and toughness, and a viewpoint which avoids the film (layer) exfoliation based on the coefficient-of-thermal-expansion difference of quartz glass and SiC. Especially when you need a member with high reinforcement, it is desirable that it is the structure which SiC permeated to the core of a member Plastic solid.

[0026] Above SiC and Si<sub>3</sub>N<sub>4</sub> etc. -- as an approach of covering the ceramics on the front face of a porosity quartz-glass object Although it is not limited especially if it is the approach of forming the film (layer) in the front face of this porosity quartz-glass object, as an especially suitable approach for the above-mentioned ceramic enveloping layer formation in this invention The CVD forming-membranes method (chemical-vapor-deposition method) which can form the high grade film and is easy to form the above-mentioned inner layer part can be mentioned. The CVD forming-membranes method is the approach of supplying a deposition raw material in the state of a gaseous phase (gas), and forming a thin film in a base material front face using a chemical reaction, and has a heat CVD method, a plasma-CVD method, an optical CVD method, etc. by with what kind of means the energy which produces a reaction is given. In this invention, any CVD method is usable.

[0027] If the standard condition is stated to a porosity quartz-glass object (disc-like base material with 20mm [ in thickness ], and a diameter of 100mm) about the case where covering formation of the SiC film is carried



out, using a heat CVD method as an example. Usually, whenever [ furnace temperature ]; on the processing conditions of 1230 degrees C, inflow gas; SiCl<sub>4</sub> (2.7SLM), C three H<sub>8</sub> (0.9SLM), H<sub>2</sub> (20SLM), and processing-time; 5-hour \*\* The covering film formation whose thickness of said film surface part which consists of SiC singleness substantially is about 100 micrometers is possible (the part of the nature foam of silica glass remains in about 3mm in thickness of a SiC infiltration inner layer part, and the core inside it).

[0028] In ceramic film (layer) covering down stream processing by this CVD method, taking procedure, like it is more desirable to divide into multiple times and to carry out film (layer) deposition processing until it becomes predetermined thickness, for example, it divides the above-mentioned processing time into 2 times by a unit of 2.5 hour, and sandwiches fluoric acid washing of a base material and desiccation actuation between them is recommended. For example, if a porosity quartz-glass object tends to be set and it is going to form the surface part of the ceramic layer of desired thickness (for example, 500-1000 micrometers) with 1 time of chemical vapor deposition on support. In case it will be in the condition that the suitable ceramic film was formed also in the part which touches the support of a porosity quartz-glass object, and support was joined to the porosity quartz-glass object and a porosity quartz-glass object is \*\*\*\*(ed), the fault which this vitreous humour itself damages will arise. Moreover, although it does not damage, the ceramic film (layer) will not fully be formed in the part which was in contact with said support, but gas impermeability will become inadequate. As described above, by dividing into multiple times and carrying out, it cannot leave the marks of the base material installation fixture in a furnace, and the film (layer) of homogeneity thickness can be formed all over the front face of a ceramic covering quartz-glass member.

[0029] In the ceramic covering quartz-glass object of this invention, suitable porosity quartz-glass voxel material is prepared, the configuration of a request of this is processed, and it considers as a base material. Subsequently, installation maintenance is carried out on a maintenance fixture, and this base material is set in a CVD furnace. And a ceramic layer is formed in the bottom of the above-mentioned condition by CVD vapor growth, for example. If it \*\*\*\* and contamination of a covering ceramic layer surface part is removed by fluoric acid washing etc. the back, it will consist of a high grade ambient atmosphere in a semi-conductor manufacture process usable suitably.

[0030] Thus, as ceramic covering film, such as formed SiC, is shown in drawing 1, a front face to the depth 10 thru/or 1000 micrometers consist only of ceramics, such as SiC, and it becomes a deep part and the ceramic [ as for the parts of 3 thru/or 5mm\*\* ] infiltration organization with which ceramics, such as SiC, usually permeated the interior of pore (air bubbles) preferably 500 micrometers or more less than 20mm from the front face from it. Usually, although the core of a base material remains with a porosity quartz-glass object, it can also consider as the above-mentioned ceramic infiltration structure to a core, and this member will become hard with high intensity. Although the rate of making the core of this base material remaining with a porosity quartz-glass object can be suitably set up by that application, in order to secure adiathermic [ as the thermal shield material for semi-conductors, and a heat insulator / the higher lightweight nature and adiathermic / higher ] especially, it is desirable to consider as 50 to 80 volume [ of the whole ceramic covering quartz-glass object ] %.

[0031] Since the above-mentioned ceramic covering quartz-glass object uses the porosity quartz-glass object for the base material, it is lightweight and is higher than ceramic independent articles, such as SiC. [ of heat insulation property ] In order to raise these engine performance more, it is desirable to use especially the latter among the nature porous body of a silica mentioned above as an example of a porosity quartz-glass object and the nature foam of silica glass. By forming ceramic layers (film), such as SiC, the raising dust of a porosity quartz-glass object can be controlled. In the case of the ceramic layer by which CVD membrane formation was carried out as the surface described above especially, it is a high grade and impurity diffusion checked is high. Moreover, chemical resistance of a surface is high because of quality of the corrosion-resistant ceramics, such as SiC, and it is possible to carry out acid cleaning of the whole member. In addition, as long as a base material is processible from tabular to the shape of a circle, and a still more complicated configuration, that the member of the configuration of arbitration can be manufactured etc. also has an advantage.

[0032] If it is in the ceramic covering quartz-glass object of this invention, as shown in drawing 2, the porosity quartz-glass layer 3 may be formed in the base material front face of the quartz-glass object 4 of arbitration, and ceramic film (layer), such as SiC, may be covered for this front face. The ceramic covering quartz-glass object of this mode is the transparence of non-air bubbles or apparent density 2.0 - 2.19 g/cm<sup>3</sup> substantially. The porosity quartz-glass layer 3 is joined to the front face of the opaque quartz-glass base material 4 with means, such as welding, and it is obtained by forming ceramic layers (1 2), such as SiC, like the above on the front face



of this porosity quartz-glass layer 3.

[0033] A porosity quartz-glass layer joins the above mentioned porosity quartz-glass object by welding etc., and although the thickness of the porosity quartz-glass layer 3 to join is suitably set up according to the configuration of a member, an application, etc., it is desirable to set it as 1 thru/or the thickness of about 10mm in the case of the usual plate. When the thickness of said porosity quartz-glass layer is thin, the joined porosity quartz-glass layer serves as the inner layer part 2 of ceramic infiltration organization, and the layer of only porosity quartz glass is lost. Therefore, although it is hard and grows into high intensity, it becomes the structure which lacks toughness and flexibility a little.

[0034] A porosity quartz-glass layer can turn into a buffer coat, and the ceramic covering quartz-glass object of this mode can prevent film peeling resulting from the coefficient-of-thermal-expansion difference between a quartz-glass object and ceramic covering film, such as SiC, and can control raising dust. Moreover, it is easily joinable with welding etc., and since both adhesion is also good, junction in a quartz-glass object and a porosity quartz-glass layer serves as a laminating structural member strong as a whole. Moreover, when the surface of a ceramic covering quartz-glass object is covered with high grade ceramics, such as SiC, it excels in impurity diffusion checked. Moreover, corrosion resistance is also high, therefore it also has many advantages the number of grades which can also carry out acid cleaning of the whole member. The ceramic covering quartz-glass object of this mode has high usefulness as high high intensity structure material of thermal insulation nature.

[0035]

[Example] the nature foam of "example 1" silica glass (apparent-density 0.4 g/cm<sup>3</sup> --) 200-800 micrometers of diameters of air bubbles, mean-coefficient-of-linear-expansion  $5.6 \times 10^{-7}/\text{degree C}$  (25-900 degrees C), Thermal conductivity 0.08 kcal/mhK, purity analysis value (ppm):Na<0.2, K< 0.2, Li<0.2, Mg<0.2, aluminum<1, and Cu<0.1 are processed into a heat-insulating-board configuration (3mm in diameter [ of 200mm ] x thickness). On this processed nature foam front face of silica glass, 60-micrometer (however, thickness of surface part which consists of SiC singleness substantially) formation of the SiC film was carried out all over the CVD furnace. In addition, CVD film growth processing conditions are; 1230 degree C and inflow gas; SiCl<sub>4</sub> whenever [ furnace temperature ]. 2.7SLM(s) and C<sub>3</sub> H<sub>8</sub> 0.9SLM(s) and H<sub>2</sub> It was referred to as 20SLM(s) and processing-time 2.5hrs. It \*\*\*\*(ed) after film formation and was used as a heat insulating board as it is after washing / desiccation. While this product showed high adiabatic efficiency, also in 80 repeat heat cycle (duration-of-service two months, seven counts of acid cleaning) use for ordinary temperature and about 800 degrees C, the raising dust from a surface was hardly seen.

[0036] The same nature porous body of a silica as the "example 2" example 1 (apparent density: 1.85g [/cm ] 3, 20-180 micrometers of pore distribution) was processed in the shape of an incubation cartridge, and 100 micrometers (however, thickness of the surface part which consists of SiC singleness substantially) were formed in the nature porous body of a silica of the shape of this incubation cartridge for the SiC film at the CVD furnace. It \*\*\*\*(ed) and was used as a heat insulating mould as it is after washing / desiccation. While the surface high grade SiC layer also enabled use of a high grade ambient atmosphere, it was equal to the corrosion by the controlled atmosphere, and there was almost no raising dust from a surface in spite of the long-term use for two months or more. Moreover, the polish recon film adheres to a front face during use of this heat insulating mould, and it is HF+HNO<sub>3</sub>. Although washed 10 times by the penetrant remover, there was no consumption by etching substantially and there were also no faults, such as shakiness.

[0037] Apparent-density 2.10 g/cm<sup>3</sup> tabular [ for "example 3" furnace walls ] An opaque quartz-glass object is prepared (3mm in 200mm[ 100mm by ] x thickness). the nature foam of silica glass with a thickness of 10mm was welded to all the front faces of this quartz-glass object (foaming quartz material -- description: -- consistency 0.8 g/cm<sup>3</sup> --) 50-200 micrometers of diameters of air bubbles, mean-coefficient-of-linear-expansion  $5.6 \times 10^{-7}/\text{degree C}$  (25-900 degrees C), thermal conductivity 0.08 kcal/mhK, purity analysis value (ppm):Na<0.2, K< 0.2, Li<0.2, Mg<0.2, aluminum<1, Cu<0.1. The SiC film was formed in the front face of this silica glass foam layer (porosity quartz-glass layer) of the opaque quartz-glass object which joined the above-mentioned nature foam of silica glass to the front face at the CVD furnace at the thickness of 300 micrometers, and it \*\*\*\*(ed) after processing, and was used as it is after washing / desiccation. The adhesion between layers of the product of an opaque quartz-glass base material, a porosity quartz-glass interlayer, and SiC surface layer each class was good, and surface exfoliation etc. was not produced in prolonged use, either.

[0038] The nature foam of silica glass with a thickness of 3mm (thing of the same description as an example 3)

was welded to all the surface sections of the transparent-silica-glass object of an "example 4" coil configuration (308mm of appearances, thickness of 311mm, die length of 350mm). 100 micrometers of SiC film were formed in this nature foam (porosity quartz-glass layer) front face of silica glass of this coil at the CVD furnace. It \*\*\*\* (ed) and was used as it is after washing desiccation. The surface high grade SiC layer was equal to the corrosion by the controlled atmosphere, while also enabling use under a high grade ambient atmosphere, and the long-term use for two months or more was possible. Moreover, it is HF+HNO<sub>3</sub> while using this coil. Although washed 10 times by the penetrant remover, there was no consumption by etching substantially and there were also no un-arranging, such as deformation.

[0039]

[Effect of the Invention] According to the ceramic covering quartz-glass object of this invention, even if it receives heat cycles, such as heating cooling, over a long period of time, exfoliation of the ceramic layer (film) which originates in the coefficient-of-thermal-expansion difference of quartz glass and a ceramic layer (film), and is produced, crack initiation, etc. can be prevented. Therefore, even if it uses the ceramic covering quartz-glass object of this invention for a long period of time under the environment where severe heating and heat-of-cooling SAIKURU \*\* are received, it deteriorates, and the raising dust of it is not carried out from a member front face, and it can be especially used suitably as a member for semi-conductor manufacture processes.

[0040] Moreover, the member which covered ceramic layers, such as SiC, on the porosity quartz-glass object among the ceramic covering quartz-glass objects of this invention is lightweight, and since it is excellent in adiathermic, it is suitable as heat insulation members, such as a heat insulating board, a thermal shield plate, and a heat insulating mould. Moreover, welding junction of the porosity quartz-glass layer is carried out, and the member of the mode in which the ceramic layer was formed on this porosity quartz-glass layer front face is excellent in tough nature, corrosion resistance, and abrasion resistance, and is used for a quartz-glass object suitable for the various members for the susceptor for furnace wall material, a coil, wafer installation, and heat treatment, the object for lamp heating apparatus, or field-like heater heating apparatus etc.

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[Translation done.]

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TECHNICAL FIELD

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[Field of the Invention] This invention relates to tough and the ceramic covering quartz-glass objects in which the ceramic layer excellent in corrosion resistance and abrasion resistance was formed, such as silicon carbide and silicon nitride, about a ceramic covering quartz-glass object on the front face of a porosity quartz-glass layer established in the front face of the porosity quartz-glass object which reinforcement \*\*\*\* in a detail easily weakly and carries out raising dust to it more, or the quartz-glass body surface.

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PRIOR ART

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[Description of the Prior Art] In the semi-conductor manufacture process, various kinds of members which consist of a porosity quartz-glass object or a quartz-glass object are used widely. For example, porosity quartz-glass objects, such as foaming quartz glass (nature foam of silica glass), are widely used in the various furnaces for semi-conductor processing as structure heat insulators, such as a heat insulating board and a wallplate, functional heat insulators, such as a wafer boat table, etc. Moreover, the quartz-glass object is widely used for the tubular Plastic solid of tabular Plastic solids, such as a thermal shield plate, a heat-resistant nozzle, a coil, etc., etc., and the wafer boat regardless of the transparent body and the opaque body.

[0003] Said porosity quartz-glass object is lightweight compared with a quartz-glass object, thermal conductivity is low, and it has the property which was excellent as a heat insulator. However, it being difficult to use a general-purpose article as it is in the semi-conductor manufacture process field which dislikes contamination according to dust in order to \*\*\*\* the usual porosity quartz-glass object easily and to carry out raising dust to the degree of pole, and quartz glass's usually enclosing a porosity quartz-glass object, etc. and a certain raising dust control measures are taken, and it is used as after, a heat insulator, etc.

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**EFFECT OF THE INVENTION**

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[Effect of the Invention] According to the ceramic covering quartz-glass object of this invention, even if it receives heat cycles, such as heating cooling, over a long period of time, exfoliation of the ceramic layer (film) which originates in the coefficient-of-thermal-expansion difference of quartz glass and a ceramic layer (film), and is produced, crack initiation, etc. can be prevented. Therefore, even if it uses the ceramic covering quartz-glass object of this invention for a long period of time under the environment where severe heating and heat-of-cooling SAIKURU \*\* are received, it deteriorates, and the raising dust of it is not carried out from a member front face, and it can be especially used suitably as a member for semi-conductor manufacture processes.

[0040] Moreover, the member which covered ceramic layers, such as SiC, on the porosity quartz-glass object among the ceramic covering quartz-glass objects of this invention is lightweight, and since it is excellent in adiathermic, it is suitable as heat insulation members, such as a heat insulating board, a thermal shield plate, and a heat insulating mould. Moreover, welding junction of the porosity quartz-glass layer is carried out, and the member of the mode in which the ceramic layer was formed on this porosity quartz-glass layer front face is excellent in tough nature, corrosion resistance, and abrasion resistance, and is used for a quartz-glass object suitable for the various members for the susceptor for furnace wall material, a coil, wafer installation, and heat treatment, the object for lamp heating apparatus, or field-like heater heating apparatus etc.

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## TECHNICAL PROBLEM

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[Problem(s) to be Solved by the Invention] By the way, the quartz-glass member which enclosed the porosity quartz-glass object, or the member for semi-conductor manufacture processes which consists of a quartz-glass object is washed using acid cleaning liquid, such as a fluoric acid water solution, in case it is used for defecation. Therefore, when it was used to some extent, by said washing, etching might progress and shakiness and breakage might take place between members. In order to solve this problem (i.e., in order to raise the chemical resistance (chemical resistance) of the member which consists of a quartz-glass object), tough and the researches and developments covered with the ceramic layer excellent in corrosion resistance and abrasion resistance of silicon carbide (SiC), silicon nitride (Si<sub>3</sub>N<sub>4</sub>), etc. are furthered in the front face.

[0005] however, when covering formation of the ceramic layers, such as the direct above-mentioned silicon carbide and silicon nitride, is carried out on the front face of a quartz-glass object Since there is a difference of the coefficient of thermal expansion ( $5.6 \times 10^{-7}/\text{degree C}$ ) of a quartz-glass object and the coefficient of thermal expansion ( $4 \times 10^{-6}/\text{degree C}$ ) of ceramics, such as said silicon carbide, When the heat histories, such as repeat heating and cooling, were received, the film (layer) destruction and exfoliation resulting from this thermal shock distortion might arise, and it might become the cause of dust.

[0006] The approach of controlling generating of such dust etc. is also proposed. To JP,8-83835,A In the quartz-glass base material which carried out surface coating with silicon carbide between this quartz-glass base material front face and a silicon carbide membrane layer For example, like silicon and silicon nitride, the inclination layer of the addition component which consists of the matter also with small degree of hardness and elastic modulus rather than silicon carbide is made to intervene as an interlayer, and the silicon carbide covering quartz-glass object which buffered heat distortion generating which originates in the difference of said thermal expansion by this interlayer is proposed. The silicon carbide covering quartz-glass object of the above-mentioned structure has good thermal shock resistance, and has the resistance stabilized in the long run about the chemical resistance force over a fluoric acid water solution, nitric-acid water solutions, these mixed-acid water solutions, etc., and dust raising dust control and diffusion evasion of semi-conductor harmful matter.

[0007] However, when the 3rd component other than a quartz-glass object and a silicon carbide component, especially a silicon ingredient were included in this way, and there was an increment in weight at this rate and it used as various members of the member for semi-conductor manufacture processes, the difficulty was in handling nature. Moreover, in order that heat capacity might increase-ize, when it used as a heat treatment member, there was a trouble that heat responsibility was not enough etc.

[0008] This invention aims at offering the ceramic covering quartz-glass object which does not have exfoliation or breakage of a ceramic layer even if it receives the thermal shock of a repeat, and raising dust, such as dust, was controlled, and was moreover excellent in chemical resistance (chemical resistance), and was excellent in handling nature.

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MEANS

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[Means for Solving the Problem] The ceramic covering quartz-glass object concerning this invention made in order to solve the above-mentioned technical technical problem is characterized by covering with a ceramic layer the front face of a porosity quartz-glass object, or the front face of a porosity quartz-glass layer established in the quartz-glass object. It is desirable for said ceramic layer to consist of silicon carbide or silicon nitride here, and it is desirable that it is the ceramic layer in which said ceramic layer was formed by the chemical-vapor-deposition method.

[0010] Moreover, it is desirable to consist of a surface part of gas impermeability which said ceramic layer becomes from ceramic singleness substantially, and a inner layer part which has the ceramic infiltration organization in which the ceramics permeated in the detailed pore of a porosity quartz-glass object or a porosity quartz-glass layer, it is in the range whose thickness of the surface part of said ceramic layer is 10 thru/or 1000 micrometers, and it is desirable for the thickness of said inner layer part to be 500 micrometers or more.

[0011] Furthermore, the consistency of the porosity quartz-glass layer which it was desirable for the thickness of the porosity quartz-glass layer prepared in said quartz-glass body surface to be 1 thru/or 10mm, and was prepared in the consistency or quartz-glass body surface of said porosity quartz-glass object is 0.1 thru/or 1.95 g/cm<sup>3</sup>. It is desirable that it is in the range.

[0012] The ceramic covering quartz-glass object of this invention has the description on a configuration in tough and the points with which it is the ceramic layer excellent in corrosion resistance and abrasion resistance, and the front face of a porosity quartz-glass object or the front face of a porosity quartz-glass layer established in the quartz-glass object is covered, such as silicon carbide and carbonization nitrogen.

[0013] When ceramic layers, such as silicon carbide (SiC), were covered on the front face of the usual quartz-glass object which is not a porous body, as already explained, it originated in the difference of the coefficient of thermal expansion of a quartz-glass object and a ceramic layer, a crack and membrane layer exfoliation arose in the ceramic layer, and this had become causes, such as dust, on it. On the other hand, with the ceramic covering quartz-glass object of this invention, ceramic covering of SiC etc. is given to the front face of a porosity quartz-glass object, or the front face of a porosity quartz-glass layer established in the quartz-glass object, namely, a ceramic layer is formed in it.

[0014] Unlike the case of the above mentioned quartz-glass object, the ceramic layer prepared in the front face of a porosity quartz-glass layer established in the front face of a porosity quartz-glass object or the quartz-glass object permeates from an interface in the detailed pore inside a porosity quartz-glass object or a porosity quartz-glass layer. That is, while the surface part of gas impermeability which consists of ceramic singleness, such as SiC, substantially is formed on said interface, the inner layer part of the infiltration organization in which said ceramics permeated in the detailed pore of a porous body is formed in the bottom of this interface. By existence of this inner layer part, the exfoliation and breakage of the ceramic layer used as a surface accompanying the coefficient-of-thermal-expansion difference of a quartz-glass object and ceramics, such as SiC, can be prevented.

[0015] If it is in the porosity quartz-glass layer prepared in the porosity quartz-glass object of this invention, and the quartz-glass object, the quartz glass is divided by countless detailed pore, and exists as organization which the very thin septum side which divides these followed. Therefore, since it is flexible and the detailed septum of quartz glass moreover has the complicated configuration by the area of jointing with ceramic layers, such as SiC, becoming large compared with the quartz-glass object to the flow stress by a thermal shock etc., respectively as compared with the quartz-glass object, stress distribution becomes easier. direct on a quartz-glass object, even if it receives the heat history like repeat heating and cooling for a long period of time, when



ceramic layers, such as SiC, are covered also with this to said porous body which was carried out -- neither film (layer) exfoliation nor the breakage by the interface occurs like [ at the time of covering SiC etc. ], but it becomes what was extremely excellent in raising dust checked.

[0016] Moreover, in the case of a SiC layer etc., the ceramic layer of corrosion resistance is high, therefore possible also for carrying out acid cleaning of the whole member. Moreover, the effectiveness that the reinforcement of the member itself increases is also acquired by covering with tough ceramics, such as SiC.

[0017] Since especially the ceramic covering quartz-glass object that consists of the porosity quartz-glass object which covered the ceramic layer of this invention uses the porosity quartz-glass object for the base material, in addition to excelling in many effectiveness, such as the above-mentioned dust raising dust checked, impurity diffusion checked, corrosion resistance, and tough nature, it is lightweight, and is high, and does so the effectiveness which was extremely excellent as a heat insulation member for semi-conductor manufacture processes. [ of adiabatic efficiency ]

[0018] Moreover, the ceramic covering quartz-glass object which consists of the porosity quartz-glass object which covered the ceramic layer of this invention can obtain the layered product which has good adhesion easily by welding a porosity quartz-glass object to the front face of a quartz-glass object. Moreover, with the ceramic covering quartz-glass object concerning this invention which covered with the ceramic layer the front face of a porosity quartz-glass layer established especially in the quartz-glass object, a porosity quartz-glass object serves as a buffer coat, and ceramics, such as SiC, becomes thermal shock resistance from the interface of this and a porosity quartz-glass object by entering into internal pore with what was extremely excellent. And since the adhesion of a porosity quartz-glass layer and ceramic layers, such as SiC, is also good, it excels in raising dust checked, such as dust, and, moreover, a ceramic layer becomes what in the case of the high grade SiC membrane layer formed by the CVD forming-membranes method etc. was excellent also in impurity diffusion checked and was extremely excellent as a member for semi-conductor manufacture processes, as already stated.

[0019] In addition, the above-mentioned ceramic covering quartz-glass object of this invention Process the configuration of a request of a porosity quartz-glass object, set the acquired porosity quartz-glass object on the support in a furnace, and the ceramic layer of gas impermeability is made to form in this porosity quartz-glass body surface by the chemical-vapor-deposition reaction. Subsequently, after \*\*\*\*(ing) and carrying out acid cleaning, it sets at the time of the front ceramic stratification. It is desirable to support a different part from the part which was in contact with said support of said porosity quartz-glass object with the support in a furnace, and to manufacture by forming the surface part of the ceramic layer of request thickness by the chemical-vapor-deposition reaction further.

[0020] For example, if a porosity quartz-glass object tends to be set and it is going to form the surface part of the ceramic layer of desired thickness (for example, 500-1000 micrometers) with 1 time of chemical vapor deposition on the support supported three points In case it will be in the condition that the suitable ceramic film was formed also in the part which touches the support of a porosity quartz-glass object, and support was joined to the porosity quartz-glass object and a porosity quartz-glass object is \*\*\*\*(ed), the fault which this vitreous humour itself damages will arise. Moreover, although it does not damage, the ceramic film (layer) will not fully be formed in the part which was in contact with said support, but gas impermeability will become inadequate.

[0021] Moreover, when a base material is a quartz-glass object, a porosity quartz-glass layer is joined to the front face of the quartz-glass object into which the desired configuration was processed by welding. Form, set said acquired quartz-glass object on the support in a furnace, and a chemical-vapor-deposition reaction is made to perform. In the time of the front [ after making the ceramic layer of gas impermeability form in this porosity quartz-glass layer front face, \*\*\*\*(ing) subsequently and carrying out acid cleaning ] ceramic stratification It is desirable from the same reason as the above to support a different part from the part which was in contact with said support of said quartz-glass object with the support in a furnace, and to manufacture by forming the surface part of the ceramic layer of request thickness by the chemical-vapor-deposition reaction further.

[0022]

[Embodiment of the Invention] This invention is explained more below at a detail. The ceramic covering quartz-glass object of this invention is the description on a configuration of the point that the front face of a porosity quartz-glass object or the front face of a porosity quartz-glass layer established in the quartz-glass object is covered with the ceramic layer. It is possible for a porosity quartz-glass object to use a material equivalent to usual quartz glass as a raw material, for it to be processed and obtained and for thermal resistance,

chemical stability, purity, etc. to lightweight-ize apparent density for this to about about 1 of usual quartz glass / 10 generally, according to quartz glass porosity-ization. And said porosity quartz-glass object is excellent also in those with low-fever expansibility, and workability with high thermal resistance. The nature porous body of a silica (apparent density: 1.00 - 1.99 g/cm<sup>3</sup>, preferably 1.80 - 1.95 g/cm<sup>3</sup>) and the nature foam of silica glass (apparent density: 0.1 - 0.99 g/cm<sup>3</sup>, preferably 0.1 - 0.50 g/cm<sup>3</sup>) which indicate below the porosity quartz-glass object which can be used by this invention are included.

[0023] Namely, are shown in JP,4-202070,A as an example of such a porosity quartz-glass object. Add the amorphous silica powder of a very detailed particle size to crystalline substance silica powder, and it mixes to it. The nature porous body of a silica which it calcinated [ porous body ] after shaping, was obtained [ porous body ], carried out the partial bond of this crystal silica \*\*\*\* for the crystalline substance silica grain front face densely by the wrap amorphous silica impalpable powder layer, and made many open pores form between crystal silica \*\*\*\*, Moreover, contain the hydroxyl group shown in JP,5-345636,A, and specific surface area heat-treats the amorphous silica base material 6m<sup>2</sup> / more than g in the ambient atmosphere containing 600 thru/or 1300-degree C ammonia gas. Subsequently, the nature foam of high grade silica glass which was made to carry out heating foaming and was obtained in the temperature requirement (1350 degrees C thru/or 1800 degrees C), Furthermore, specified quantity addition of the high grade silicon nitride by which particle-size control was carried out at the nature raw material powder of high grade silicic acid shown in JP,5-254882,A is carried out, and the bubble diameter distribution 20 thru/or 180-micrometer nature foam of silica glass, etc. obtained by fusing by the oxyhydrogen flame is mentioned. It sets to this invention also especially among these porosity quartz-glass objects, and they are a consistency 0.1 thru/or 1.95 g/cm<sup>3</sup>. It is desirable from a viewpoint of contamination tightness, lightweight nature, adiathermic, and heat-capacity balance to use a high grade article.

[0024] In this invention, surface coating of the front face of the above-mentioned porosity quartz-glass object is carried out by tough and the ceramic film (layer) excellent in corrosion resistance and abrasion resistance, such as silicon carbide (SiC) and silicon nitride (Si<sub>3</sub>N<sub>4</sub>). SiC and Si<sub>3</sub>N<sub>4</sub> which are formed in the front face of this porosity quartz-glass object etc. -- as shown in drawing 1 as a mimetic diagram, as for the film (layer), it is desirable to consist of the surface part 1 of gas impermeability which membranous cross-section organization becomes from ceramic singleness substantially, and the inner layer part 2 of the infiltration organization in which the ceramics permeated in the detailed pore of the porosity quartz-glass object 3.

[0025] And if it is in the ceramic covering quartz-glass object which carried out SiC covering of the porosity quartz-glass object again, as for the thickness of the gas impermeable surface section 1 of said SiC singleness, it is desirable from a chemical-resistant and dust raising dust checked point that it is in 10 thru/or the range of 1000 micrometers. Moreover, as for the thickness of the inner layer part 2 which has the SiC infiltration organization in which SiC permeated in the detailed pore of said porosity quartz-glass object, it is desirable that it is 3mm or more from the viewpoint which collateralizes reinforcement and toughness, and a viewpoint which avoids the film (layer) exfoliation based on the coefficient-of-thermal-expansion difference of quartz glass and SiC. Especially when you need a member with high reinforcement, it is desirable that it is the structure which SiC permeated to the core of a member Plastic solid.

[0026] Above SiC and Si<sub>3</sub>N<sub>4</sub> etc. -- as an approach of covering the ceramics on the front face of a porosity quartz-glass object Although it is not limited especially if it is the approach of forming the film (layer) in the front face of this porosity quartz-glass object, as an especially suitable approach for the above-mentioned ceramic enveloping layer formation in this invention The CVD forming-membranes method (chemical-vapor-deposition method) which can form the high grade film and is easy to form the above-mentioned inner layer part can be mentioned. The CVD forming-membranes method is the approach of supplying a deposition raw material in the state of a gaseous phase (gas), and forming a thin film in a base material front face using a chemical reaction, and has a heat CVD method, a plasma-CVD method, an optical CVD method, etc. by with what kind of means the energy which produces a reaction is given. In this invention, any CVD method is usable.

[0027] If the standard condition is stated to a porosity quartz-glass object (disc-like base material with 20mm [ in thickness ], and a diameter of 100mm) about the case where covering formation of the SiC film is carried out, using a heat CVD method as an example Usually, whenever [ furnace temperature ]; on the processing conditions of 1230 degrees C, inflow gas; SiCl<sub>4</sub> (2.7SLM), C three H<sub>8</sub> (0.9SLM), H<sub>2</sub> (20SLM), and processing-time; 5-hour \*\* The covering film formation whose thickness of said film surface part which consists of SiC

singleness substantially is about 100 micrometers is possible (the part of the nature foam of silica glass remains in about 3mm in thickness of a SiC infiltration inner layer part, and the core inside it).

[0028] In ceramic film (layer) covering down stream processing by this CVD method, taking procedure, like it is more desirable to divide into multiple times and to carry out film (layer) deposition processing until it becomes predetermined thickness, for example, it divides the above-mentioned processing time into 2 times by a unit of 2.5 hour, and sandwiches fluoric acid washing of a base material and desiccation actuation between them is recommended. For example, if a porosity quartz-glass object tends to be set and it is going to form the surface part of the ceramic layer of desired thickness (for example, 500-1000 micrometers) with 1 time of chemical vapor deposition on support. In case it will be in the condition that the suitable ceramic film was formed also in the part which touches the support of a porosity quartz-glass object, and support was joined to the porosity quartz-glass object and a porosity quartz-glass object is \*\*\*\*(ed), the fault which this vitreous humour itself damages will arise. Moreover, although it does not damage, the ceramic film (layer) will not fully be formed in the part which was in contact with said support, but gas impermeability will become inadequate. As described above, by dividing into multiple times and carrying out, it cannot leave the marks of the base material installation fixture in a furnace, and the film (layer) of homogeneity thickness can be formed all over the front face of a ceramic covering quartz-glass member.

[0029] In the ceramic covering quartz-glass object of this invention, suitable porosity quartz-glass voxel material is prepared, the configuration of a request of this is processed, and it considers as a base material. Subsequently, installation maintenance is carried out on a maintenance fixture, and this base material is set in a CVD furnace. And a ceramic layer is formed in the bottom of the above-mentioned condition by CVD vapor growth, for example. If it \*\*\*\* and contamination of a covering ceramic layer surface part is removed by fluoric acid washing etc. the back, it will consist of a high grade ambient atmosphere in a semi-conductor manufacture process usable suitably.

[0030] Thus, as ceramic covering film, such as formed SiC, is shown in drawing 1, a front face to the depth 10 thru/or 1000 micrometers consist only of ceramics, such as SiC, and it becomes a deep part and the ceramic [ as for the parts of 3 thru/or 5mm\*\* ] infiltration organization with which ceramics, such as SiC, usually permeated the interior of pore (air bubbles) preferably 500 micrometers or more less than 20mm from the front face from it. Usually, although the core of a base material remains with a porosity quartz-glass object, it can also consider as the above-mentioned ceramic infiltration structure to a core, and this member will become hard with high intensity. Although the rate of making the core of this base material remaining with a porosity quartz-glass object can be suitably set up by that application, in order to secure adiathermic [ as the thermal shield material for semi-conductors, and a heat insulator / the higher lightweight nature and adiathermic / higher ] especially, it is desirable to consider as 50 to 80 volume [ of the whole ceramic covering quartz-glass object ] %.

[0031] Since the above-mentioned ceramic covering quartz-glass object uses the porosity quartz-glass object for the base material, it is lightweight and is higher than ceramic independent articles, such as SiC. [ of heat insulation property ] In order to raise these engine performance more, it is desirable to use especially the latter among the nature porous body of a silica mentioned above as an example of a porosity quartz-glass object and the nature foam of silica glass. By forming ceramic layers (film), such as SiC, the raising dust of a porosity quartz-glass object can be controlled. In the case of the ceramic layer by which CVD membrane formation was carried out as the surface described above especially, it is a high grade and impurity diffusion checked is high. Moreover, chemical resistance of a surface is high because of quality of the corrosion-resistant ceramics, such as SiC, and it is possible to carry out acid cleaning of the whole member. In addition, as long as a base material is processible from tabular to the shape of a circle, and a still more complicated configuration, that the member of the configuration of arbitration can be manufactured etc. also has an advantage.

[0032] If it is in the ceramic covering quartz-glass object of this invention, as shown in drawing 2, the porosity quartz-glass layer 3 may be formed in the base material front face of the quartz-glass object 4 of arbitration, and ceramic film (layer), such as SiC, may be covered for this front face. The ceramic covering quartz-glass object of this mode is the transparency of non-air bubbles or apparent density 2.0 - 2.19 g/cm<sup>3</sup> substantially. The porosity quartz-glass layer 3 is joined to the front face of the opaque quartz-glass base material 4 with means, such as welding, and it is obtained by forming ceramic layers (1 2), such as SiC, like the above on the front face of this porosity quartz-glass layer 3.

[0033] A porosity quartz-glass layer joins the above mentioned porosity quartz-glass object by welding etc., and although the thickness of the porosity quartz-glass layer 3 to join is suitably set up according to the

configuration of a member, an application, etc., it is desirable to set it as 1 thru/or the thickness of about 10mm in the case of the usual plate. When the thickness of said porosity quartz-glass layer is thin, the joined porosity quartz-glass layer serves as the inner layer part 2 of ceramic infiltration organization, and the layer of only porosity quartz glass is lost. Therefore, although it is hard and grows into high intensity, it becomes the structure which lacks toughness and flexibility a little.

[0034] A porosity quartz-glass layer can turn into a buffer coat, and the ceramic covering quartz-glass object of this mode can prevent film peeling resulting from the coefficient-of-thermal-expansion difference between a quartz-glass object and ceramic covering film, such as SiC, and can control raising dust. Moreover, it is easily joinable with welding etc., and since both adhesion is also good, junction in a quartz-glass object and a porosity quartz-glass layer serves as a laminating structural member strong as a whole. Moreover, when the surface of a ceramic covering quartz-glass object is covered with high grade ceramics, such as SiC, it excels in impurity diffusion checked. Moreover, corrosion resistance is also high, therefore it also has many advantages the number of grades which can also carry out acid cleaning of the whole member. The ceramic covering quartz-glass object of this mode has high usefulness as high high intensity structure material of thermal insulation nature.

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[Translation done.]

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EXAMPLE

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[Example] Nature foam of "example 1" silica glass (apparent-density 0.4 g/cm<sup>3</sup>, 200-800 micrometers of diameters of air bubbles, mean-coefficient-of-linear-expansion  $5.6 \times 10^{-7}$ /degree C (25-900 degrees C)) Thermal conductivity 0.08 kcal/mhK, purity analysis value (ppm):Na<0.2, K<0.2, Li<0.2, Mg<0.2, aluminum<1, and Cu<0.1 are processed into a heat-insulating-board configuration (3mm in diameter [ of 200mm ] x thickness). On this processed nature foam front face of silica glass, 60-micrometer (however, thickness of surface part which consists of SiC singleness substantially) formation of the SiC film was carried out all over the CVD furnace. In addition, CVD film growth processing conditions are; 1230 degree C and inflow gas; SiCl<sub>4</sub> whenever [ furnace temperature ]. 2.7SLM(s) and C<sub>3</sub>H<sub>8</sub> 0.9SLM(s) and H<sub>2</sub> It was referred to as 20SLM(s) and processing-time 2.5hrs. It \*\*\*\*(ed) after film formation and was used as a heat insulating board as it is after washing / desiccation. While this product showed high adiabatic efficiency, also in 80 repeat heat cycle (duration-of-service two months, seven counts of acid cleaning) use for ordinary temperature and about 800 degrees C, the raising dust from a surface was hardly seen.

[0036] The same nature porous body of a silica as the "example 2" example 1 (apparent density: 1.85g [/cm ]<sup>3</sup>, 20-180 micrometers of pore distribution) was processed in the shape of an incubation cartridge, and 100 micrometers (however, thickness of the surface part which consists of SiC singleness substantially) were formed in the nature porous body of a silica of the shape of this incubation cartridge for the SiC film at the CVD furnace. It \*\*\*\*(ed) and was used as a heat insulating mould as it is after washing / desiccation. While the surface high grade SiC layer also enabled use of a high grade ambient atmosphere, it was equal to the corrosion by the controlled atmosphere, and there was almost no raising dust from a surface in spite of the long-term use for two months or more. Moreover, the polish recon film adheres to a front face during use of this heat insulating mould, and it is HF+HNO<sub>3</sub>. Although washed 10 times by the penetrant remover, there was no consumption by etching substantially and there were also no faults, such as shakiness.

[0037] Apparent-density 2.10 g/cm<sup>3</sup> tabular [ for "example 3" furnace walls ] An opaque quartz-glass object is prepared (3mm in 200mm[ 100mm by ] x thickness), the nature foam of silica glass with a thickness of 10mm was welded to all the front faces of this quartz-glass object (foaming quartz material -- description: -- consistency 0.8 g/cm<sup>3</sup> --) 50-200 micrometers of diameters of air bubbles, mean-coefficient-of-linear-expansion  $5.6 \times 10^{-7}$ /degree C (25-900 degrees C), thermal conductivity 0.08 kcal/mhK, purity analysis value (ppm):Na<0.2, K<0.2, Li<0.2, Mg<0.2, aluminum<1, Cu<0.1. The SiC film was formed in the front face of this silica glass foam layer (porosity quartz-glass layer) of the opaque quartz-glass object which joined the above-mentioned nature foam of silica glass to the front face at the CVD furnace at the thickness of 300 micrometers, and it \*\*\*\*(ed) after processing, and was used as it is after washing / desiccation. The adhesion between layers of the product of an opaque quartz-glass base material, a porosity quartz-glass interlayer, and SiC surface layer each class was good, and surface exfoliation etc. was not produced in prolonged use, either.

[0038] The nature foam of silica glass with a thickness of 3mm (thing of the same description as an example 3) was welded to all the surface sections of the transparent-silica-glass object of an "example 4" coil configuration (308mm of appearances, thickness of 311mm, die length of 350mm). 100 micrometers of SiC film were formed in this nature foam (porosity quartz-glass layer) front face of silica glass of this coil at the CVD furnace. It \*\*\*\*(ed) and was used as it is after washing desiccation. The surface high grade SiC layer was equal to the corrosion by the controlled atmosphere, while also enabling use under a high grade ambient atmosphere, and the long-term use for two months or more was possible. Moreover, it is HF+HNO<sub>3</sub> while using this coil. Although washed 10 times by the penetrant remover, there was no consumption by etching substantially and there were also no un-arranging, such as deformation.

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DESCRIPTION OF DRAWINGS

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[Brief Description of the Drawings]

[Drawing 1] Drawing 1 is the sectional view having shown typically the organization of the ceramic covering quartz-glass object of this invention of a mode with which a base material consists of a porosity quartz-glass object.

[Drawing 2] Drawing 2 is drawing having shown typically the ceramic covering quartz-glass object of this invention of the mode in which the ceramic layer was formed on the front face which welded the porosity quartz-glass layer to the quartz-glass object, (a) is the perspective view and (b) is the partial expanded sectional view.

[Description of Notations]

- 1 SiC Enveloping Layer (Film) (Surface Part)
- 2 SiC Enveloping Layer (Film) (Inner Layer Part)
- 3 Porosity Quartz-Glass Object (Porosity Quartz-Glass Layer)
- 4 Quartz-Glass Object

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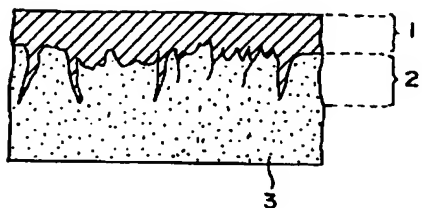
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DRAWINGS

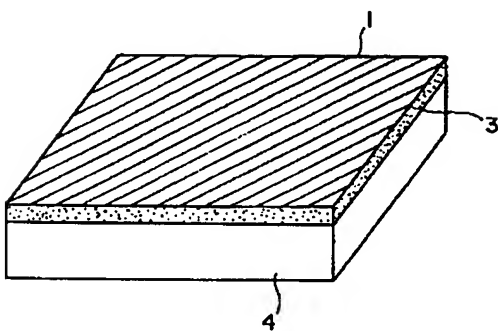
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[Drawing 1]

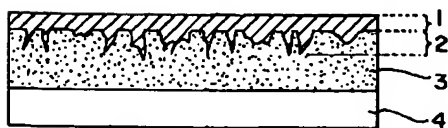


[Drawing 2]

(a)



(b)



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PAT-NO: JPC2001261375A  
DOCUMENT-IDENTIFIER: JP 2001261375 A  
TITLE: CERAMIC-COATED QUARTZ GLASS BODY  
PUBN-DATE: September 26, 2001

INVENTOR-INFORMATION:

NAME	COUNTRY
SATO, HIROMASA	N/A
TAKEDA, SHUICHI	N/A
INABA, TAKESHI	N/A
SOTODANI, EIICHI	N/A

ASSIGNEE-INFORMATION:

NAME	COUNTRY
TOSHIBA CERAMICS CO LTD	N/A

APPL-NO: JP2000070477  
APPL-DATE: March 14, 2000

INT-CL (IPC): C03C017/22, C23C016/34 , C23C016/42

ABSTRACT:

PROBLEM TO BE SOLVED: To obtain a ceramic-coated quartz glass body having neither peeling nor damage of a ceramic layer even if repeatedly subjected to thermal shocks being controlled in dust, etc., having excellent chemical resistance and excellent handleability.

SOLUTION: This ceramic-coated quartz glass body is obtained by coating the surface of a porous quartz glass body 3 or the surface a porous quartz glass layer formed on a quartz glass body with ceramic layers 1 and 2. The ceramic-coated quartz glass body is produced by forming a ceramic layer on the surface of the porous quartz glass body or the porous quartz glass layer by a chemical vapor-phase growth reaction, then taking out the porous quartz glass from a furnace and cleaning the ceramic layer with an acid.

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DERWENT-ACC-NO: 2002-109337

DERWENT-WEEK: 200215

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TITLE: Ceramic coated quartz glass for semiconductor production, has silicon carbide or silicon nitride ceramic layer formed on surface of porous quartz glass layer on quartz glass

PATENT-ASSIGNEE: TOSHIBA CERAMICS CO[TOSF]

PRIORITY-DATA: 2000JP-0070477 (March 14, 2000)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES
MAIN-IPC			
JP 2001261375 A	September 26, 2001	N/A	007
C03C 017/22			

APPLICATION-DATA:

PUB-NO	APPL-DESCRIPTOR	APPL-NO	APPL-DATE
JP2001261375A	N/A	2000JP-0070477	March 14, 2000

INT-CL (IPC): C03C017/22, C23C016/34, C23C016/42

ABSTRACTED-PUB-NO: JP2001261375A

BASIC-ABSTRACT:

NOVELTY - The ceramic coated quartz glass has a silicon carbide or silicon nitride ceramic layer formed on surface of porous quartz glass layer (3) on quartz glass.

DETAILED DESCRIPTION - The ceramic layer is formed by chemical vapor phase epitaxial method. The porous quartz layer has a density of 0.1-1.95 g/cm<sup>3</sup>, and a thickness of 1-10 mm.

USE - The ceramic coated quartz glass is used for semiconductor production, and as heat insulating board, heat shielding board and heat retention tube, susceptor for furnace wall material, reaction tube, wafer mount, heating apparatus, sheet-like heater.

ADVANTAGE - The ceramic coated quartz glass is lightweight, and has excellent thermal insulation, chemical resistance, corrosion resistance and wear resistance. Even when repeated heating and cooling of the ceramic coated quartz glass are performed, peeling of ceramic layer, and generation of crack on ceramic layer are prevented.

DESCRIPTION OF DRAWING(S) - The figure shows the sectional drawing of the ceramic coated quartz glass.

Silicon carbide coating layers 1,2

Porous quartz layer 3

CHOSEN-DRAWING: Dwg.1/2

TITLE-TERMS: CERAMIC COATING QUARTZ GLASS SEMICONDUCTOR PRODUCE SILICON CARBIDE  
SILICON NITRIDE CERAMIC LAYER FORMING SURFACE POROUS QUARTZ GLASS  
LAYER QUARTZ GLASS

DERWENT-CLASS: L01

CPI-CODES: L01-G04C; L01-L04;

SECONDARY-ACC-NO:

CPI Secondary Accession Numbers: C2002-033738

(19) 日本国特許庁 (J P)

(12) 公開特許公報 (A)

(11) 特許出願公開番号

特開2001-261375

(P2001-261375A)

(43) 公開日 平成13年9月26日 (2001.9.26)

(51) IntCl.

識別記号

F I

テロト (参考)

C 0 3 C 17/22

C 0 3 C 17/22

Z 4 G 0 5 9

// C 2 3 C 16/34

C 2 3 C 16/34

4 K 0 3 0

16/42

16/42

審査請求 未請求 請求項の数 7 O L (全 7 頁)

(21) 出願番号 特願2000-70477(P2000-70477)

(22) 出願日 平成12年3月14日 (2000.3.14)

(71) 出願人 000221122

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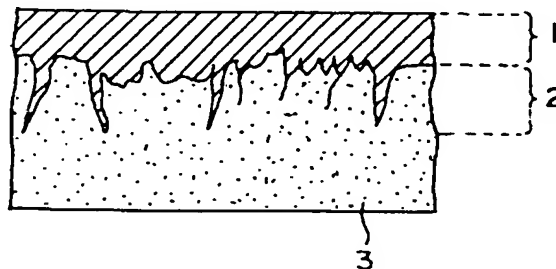
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(54) 【発明の名称】 セラミックス被覆石英ガラス体

(57) 【要約】

【課題】 繰り返しの熱衝撃を受けてもセラミックス層の剥離や破損がなく、ダスト等の発塵が抑制され、しかも化学的耐性 (耐薬品性) に優れ、かつハンドリング性に優れたセラミックス被覆石英ガラス体を提供する。

【解決手段】 セラミックス被覆石英ガラス体は、多孔質石英ガラス体3の表面が、あるいは石英ガラス体に設けられた多孔質石英ガラス層の表面がセラミックス層1、2で被覆されている。このセラミックス被覆石英ガラス体は、化学気相成長反応により該多孔質石英ガラス体、あるいは多孔質石英ガラス層の表面にセラミックス層を形成させ、次いで炉出しして酸洗浄することによって製造される。



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## 【特許請求の範囲】

【請求項1】 多孔質石英ガラス体の表面を、あるいは石英ガラス体に設けられた多孔質石英ガラス層の表面をセラミックス層で被覆したことを特徴とするセラミックス被覆石英ガラス体。

【請求項2】 前記セラミックス層が炭化珪素または窒化珪素から成ることを特徴とする請求項1記載のセラミックス被覆石英ガラス体。

【請求項3】 前記セラミックス層が化学気相成長法により形成されたセラミックス層であることを特徴とする請求項1または請求項2記載のセラミックス被覆石英ガラス体。

【請求項4】 前記セラミックス層が、実質的にセラミックス単身からなるガス不透過性の表層部分と、セラミックスが多孔質石英ガラス体あるいは多孔質石英ガラス層の微細気孔内に浸透したセラミックス浸潤組織構造を有する内層部分とから構成されていることを特徴とする請求項1乃至請求項3のいずれかに記載のセラミックス被覆石英ガラス体。

【請求項5】 前記セラミックス層の表層部分の厚さが10乃至1000 $\mu\text{m}$ の範囲にあり、前記内層部分の厚さが500 $\mu\text{m}$ 以上であることを特徴とする請求項4記載のセラミックス被覆石英ガラス体。

【請求項6】 前記石英ガラス体表面に設けられた多孔質石英ガラス層の厚さが1乃至10mmであることを特徴とする請求項1乃至請求項5のいずれかに記載のセラミックス被覆石英ガラス体。

【請求項7】 前記多孔質石英ガラス体の密度、あるいは石英ガラス体表面に設けられた多孔質石英ガラス層の密度が0.1乃至1.95 $\text{g}/\text{cm}^3$ の範囲にあることを特徴とする請求項1乃至請求項6のいずれかに記載のセラミックス被覆石英ガラス体。

## 【発明の詳細な説明】

## 【0001】

【発明の属する技術分野】本発明は、セラミックス被覆石英ガラス体に関し、より詳細には、強度が弱く容易に粉壊し発塵してしまう多孔質石英ガラス体の表面に、あるいは石英ガラス体表面に設けられた多孔質石英ガラス層の表面に、炭化珪素、窒化珪素等の強靱かつ耐食性、耐摩耗性に優れたセラミックス層を形成したセラミックス被覆石英ガラス体に関する。

## 【0002】

【従来の技術】半導体製造プロセスにおいては、多孔質石英ガラス体や石英ガラス体から成る各種の部材が広く使用されている。例えば、発泡石英ガラス（シリカガラス質発泡体）等の多孔質石英ガラス体は、半導体処理用の各種炉において、保温板や壁材等の構造断熱材等として、また、ウエハポートテーブルなどの機能性断熱材等として、広く使用されている。また、石英ガラス体は、熱遮蔽板等の板状成形体や耐熱ノズル、反応管等の管状

成形体、ウエハポートに透明体、不透明体を問わず広く使用されている。

【0003】前記多孔質石英ガラス体は石英ガラス体に比べて、軽量で、熱伝導率が低く、断熱材として優れた性質を有している。しかしながら、通常多孔質石英ガラス体は、容易に粉壊して発塵するため、粉塵による汚染を極度に嫌う半導体製造プロセス分野では、汎用品をそのまま使用することは困難であり、通常、多孔質石英ガラス体を石英ガラスで封入する等、何らかの発塵抑制処置を施して後、断熱材等として使用している。

## 【0004】

【発明が解決しようとする課題】ところで、多孔質石英ガラス体を封入した石英ガラス部材、あるいは石英ガラス体から成る半導体製造プロセス用部材は、清浄化のため、使用する際、フッ酸水溶液等の酸洗浄液を用いて洗浄される。そのため、ある程度使用すると前記洗浄によって、エッチングが進み、部材間にながつきや破損が起こることがあった。かかる問題を解決するため、即ち、石英ガラス体からなる部材の化学的耐性（耐薬品性）を向上させるため、その表面を炭化珪素（ $\text{SiC}$ ）や窒化珪素（ $\text{Si}_3\text{N}_4$ ）等の強靱かつ耐食性、耐摩耗性に優れたセラミックス層で被覆する研究開発が進められている。

【0005】しかしながら、石英ガラス体の表面に直接上記炭化珪素や窒化珪素等のセラミックス層を被覆形成した場合には、石英ガラス体の熱膨張係数（ $5.6 \times 10^{-7}/^\circ\text{C}$ ）と前記炭化珪素等のセラミックスの熱膨張係数（ $\text{SiC} : 4 \times 10^{-6}/^\circ\text{C}$ ）との差があるため、繰り返し加熱・冷却等の熱履歴を受けると、この熱衝撃歪みに起因する膜（層）破壊や剥離が生じ、ダストの原因になることがあった。

【0006】このようなダスト等の発生を抑制する方法も提案されており、例えば特開平8-83835号公報には、炭化珪素で表面被覆した石英ガラス基材において、該石英ガラス基材表面と炭化珪素膜層との間に、例えば珪素、窒化珪素等のように炭化珪素よりも硬度も弾性率も小さい物質から成る付加成分の勾配層を中間層として介在させ、この中間層により前記熱膨張の差に起因する熱歪み発生を緩和した炭化珪素被覆石英ガラス体が提案されている。上記構造の炭化珪素被覆石英ガラス体は、良好な熱衝撃抵抗性を有し、フッ酸水溶液、硝酸水溶液、これらの混酸水溶液等に対する化学抵抗力、また、ダスト発塵抑制や半導体有害物質の拡散回避に関し、長期的に安定した耐性を有している。

【0007】しかしながら、このように石英ガラス体と炭化珪素成分以外の第3の成分、特に珪素材料を含んだものであると、この分の重量増加があり、半導体製造プロセス用部材の各種部材として用いた場合、ハンドリング性に難点があった。また、熱容量が増大化するため、熱処理部材として用いた場合に熱応答性が充分でな

い等の問題点があった。

【0008】本発明は、繰り返しの熱衝撃を受けてもセラミックス層の剥離や破損がなく、ダスト等の発塵が抑制され、しかも化学的耐性（耐薬品性）に優れ、かつハンドリング性に優れたセラミックス被覆石英ガラス体を提供することを目的とするものである。

【0009】

【課題を解決するための手段】上記技術的課題を解決するためになされた本発明にかかるセラミックス被覆石英ガラス体は、多孔質石英ガラス体の表面を、あるいは石英ガラス体に設けられた多孔質石英ガラス層の表面をセラミックス層で被覆したことを特徴としている。ここで、前記セラミックス層が炭化珪素または窒化珪素から成ることが望ましく、また、前記セラミックス層が化学気相成長法により形成されたセラミックス層であることが望ましい。

【0010】また、前記セラミックス層が、実質的にセラミックス単身からなるガス不透過性の表層部分と、セラミックスが多孔質石英ガラス体あるいは多孔質石英ガラス層の微細気孔内に浸透したセラミックス浸潤組織構造を有する内層部分とから構成されていることが望ましく、前記セラミックス層の表層部分の厚さが10乃至1000 $\mu\text{m}$ の範囲にあり、前記内層部分の厚さが500 $\mu\text{m}$ 以上であることが望ましい。

【0011】更に、前記石英ガラス体表面に設けられた多孔質石英ガラス層の厚さが1乃至10mmであることが望ましく、前記多孔質石英ガラス体の密度、あるいは石英ガラス体表面に設けられた多孔質石英ガラス層の密度が0.1乃至1.95 $\text{g}/\text{cm}^3$ の範囲にあることが望ましい。

【0012】本発明のセラミックス被覆石英ガラス体は、炭化珪素、炭化窒素等の強靱かつ耐食性、耐摩耗性に優れたセラミックス層で、多孔質石英ガラス体の表面、あるいは石英ガラス体に設けられた多孔質石英ガラス層の表面が被覆されている点に構成上の特徴がある。

【0013】多孔質体でない通常の石英ガラス体の表面に、例えば、炭化珪素（SiC）等のセラミックス層を被覆した場合は、既に説明したように、石英ガラス体とセラミックス層の熱膨張係数の相違に起因して、セラミックス層にひび割れや膜層剥離が生じ、これがダスト等の原因となっていた。これに対し、本発明のセラミックス被覆石英ガラス体では、多孔質石英ガラス体の表面、あるいは石英ガラス体に設けられた多孔質石英ガラス層の表面に、SiC等のセラミックス被覆を施す、即ち、セラミックス層を形成するものである。

【0014】多孔質石英ガラス体の表面に、あるいは石英ガラス体に設けられた多孔質石英ガラス層の表面に設けられたセラミックス層は、前記した石英ガラス体の場合と異なり、界面から、多孔質石英ガラス体の内部あるいは多孔質石英ガラス層の内部の微細気孔内に浸透す

る。即ち、前記界面上には、実質的にSiC等のセラミックス単身からなるガス不透過性の表層部分が形成されると共に、該界面下に前記セラミックスが多孔質体の微細気孔内に浸透した浸潤組織構造の内層部分が形成される。この内層部分の存在により、石英ガラス体とSiC等のセラミックスの熱膨張係数差に伴う、表層となるセラミックス層の剥離や破損を防止することができる。

【0015】本発明の多孔質石英ガラス体、石英ガラス体に設けられた多孔質石英ガラス層にあっては、その石英ガラスは無数の微細気孔により分割され、これらを区画する極薄い隔壁面が連続した組織構造として存在する。従って、石英ガラス体に比較して熱衝撃等による変形応力に対し柔軟であり、しかも、石英ガラス体に比べてSiC等セラミックス層との接着部の面積が広くなり、かつ、石英ガラスの微細隔壁は夫々複雑な形状を有しているため応力分散がより容易となる。このことによっても、前記したような多孔質体にSiC等のセラミックス層を被覆した場合には、繰り返し加熱・冷却のような熱履歴を長期間受けても、石英ガラス体に直接SiC等を被覆した場合のように膜（層）剥離や界面での破損が発生せず、発塵抑制性に極めて優れたものとなる。

【0016】また、セラミックス層がSiC層等の場合には耐食性も高く、そのため部材全体を酸洗浄することも可能である。また、SiC等の強靱なセラミックスで被覆することにより、部材自体の強度が増加するという効果も得られる。

【0017】特に、本発明のセラミックス層を被覆した多孔質石英ガラス体から成るセラミックス被覆石英ガラス体は、基材に多孔質石英ガラス体を用いているため、上記したダスト発塵抑制性、不純物拡散抑制性、耐食性、強靱性等の諸効果に優れていることに加え、軽量で、かつ断熱効果も高く、半導体製造プロセス用断熱部材として極めて優れた効果を奏する。

【0018】また、本発明のセラミックス層を被覆した多孔質石英ガラス体から成るセラミックス被覆石英ガラス体は、石英ガラス体の表面に多孔質石英ガラス体を融着することによって、容易に良好な密着性を有する積層体を得ることができる。また、特に石英ガラス体に設けられた多孔質石英ガラス層の表面をセラミックス層で被覆した本発明にかかるセラミックス被覆石英ガラス体では、多孔質石英ガラス体が緩衝層となり、SiC等のセラミックスがこれと多孔質石英ガラス体との界面から内部の気孔に入り込むことで、耐熱衝撃性に極めて優れたものとなる。そして、既に述べたとおり、多孔質石英ガラス層とSiC等のセラミックス層との密着性も良好であるため、ダスト等の発塵抑制性に優れ、しかも、セラミックス層が、CVD成膜法等により形成された高純度SiC膜層の場合には、不純物拡散抑制性にも優れ、半導体製造プロセス用部材として極めて優れたものとなる。



【0019】なお、本発明の上記セラミックス被覆石英ガラス体は、多孔質石英ガラス体を所望の形状に加工し、得られた多孔質石英ガラス体を炉中の支持具上にセットして化学気相成長反応により該多孔質石英ガラス体表面にガス不透過性のセラミックス層を形成させ、次いで炉出しして酸洗浄した後、前セラミックス層形成時において、前記多孔質石英ガラス体の前記支持具と接していた部位とは異なる部位を炉中の支持具により支持し、さらに化学気相成長反応により、所望厚さのセラミックス層の表層部分を形成することにより製造することが好ましい。

【0020】例えば、3点支持する支持具上に多孔質石英ガラス体をセットし、1回の化学気相成長によって所望の厚さ（例えば500～1000 $\mu$ m）のセラミックス層の表層部分を形成しようとする、多孔質石英ガラス体の支持具と接する部分にも、相応のセラミックス膜が形成され多孔質石英ガラス体と支持具が接合された状態となり、多孔質石英ガラス体を炉出しする際に、このガラス体自身が破損してしまう不具合が生じてしまう。また、破損しないまでも前記支持具と接していた部分に十分にセラミックス膜（層）が形成されず、ガス不透過性が不十分になってしまう。

【0021】また、基材が石英ガラス体の場合は、所望の形状に加工した石英ガラス体の表面に多孔質石英ガラス層を融着により接合、形成し、得られた前記石英ガラス体を炉中の支持具上にセットして化学気相成長反応を行わせ、該多孔質石英ガラス層表面にガス不透過性のセラミックス層を形成させ、次いで炉出しして酸洗浄した後、前セラミックス層形成時において、前記石英ガラス体の前記支持具と接していた部位とは異なる部位を炉中の支持具により支持し、さらに化学気相成長反応により、所望厚さのセラミックス層の表層部分を形成することにより製造することが、上記同様の理由から好ましい。

【0022】

【発明の実施の形態】以下に本発明をより詳細に説明する。本発明のセラミックス被覆石英ガラス体は、多孔質石英ガラス体の表面が、あるいは石英ガラス体に設けられた多孔質石英ガラス層の表面がセラミックス層で被覆されている点が構成上の特徴である。一般に、多孔質石英ガラス体は、通常の石英ガラスと同等の素材を原料とし、これを多孔質化処理して得られるもので、耐熱性、化学安定性、純度等は、石英ガラスに準じ、見掛密度を通常の石英ガラスの約1/10程度まで軽量化することが可能である。そして、前記多孔質石英ガラス体は、高耐熱性で低熱膨張性あり、加工性にも優れている。本発明で用いることのできる多孔質石英ガラス体は、次に記載するシリカ質多孔体（見掛密度：1.00～1.99 $g/cm^3$ 、好ましくは、1.80～1.95 $g/cm^3$ ）やシリカガラス質発泡体（見掛密度：0.1～0.

99 $g/cm^3$ 、好ましくは、0.1～0.50 $g/cm^3$ ）が含まれる。

【0023】即ち、このような多孔質石英ガラス体の例として、特開4-202070号公報に示される、結晶質シリカ粉末に極微細な粒径の非晶質シリカ粉末を添加し、混合、成形後焼成して得られ、結晶質シリカ粒表面を覆う非晶質シリカ微粉末層により該結晶シリカ質粒を密に部分結合させて結晶シリカ質粒間に多数の開気孔を形成させたシリカ質多孔体、また特開平5-345636号公報に示される、水酸基を含有し、比表面積が6 $m^2/g$ 以上の非晶質シリカ母材を600乃至1300 $^{\circ}C$ のアンモニアガスを含んだ雰囲気中で熱処理し、次いで1350 $^{\circ}C$ 乃至1800 $^{\circ}C$ の温度範囲で加熱発泡させて得られた高純度シリカガラス質発泡体、更には特開平5-254882号公報に示される高純度珪酸質原料粉に粒径制御された高純度窒化珪素を所定量添加し、酸水素炎で熔融して得られた泡径分布20乃至180 $\mu$ mのシリカガラス質発泡体等が挙げられる。これらの多孔質石英ガラス体の内でも特に、本発明においては、密度0.1乃至1.95 $g/cm^3$ の高純度品を用いることが、汚染防止性、軽量性、断熱性、熱容量バランスの観点から好ましい。

【0024】本発明においては、上記の多孔質石英ガラス体の表面を炭化珪素（SiC）、窒化珪素（Si<sub>3</sub>N<sub>4</sub>）等の強靱かつ耐食性、耐摩耗性に優れたセラミックス膜（層）で表面被覆する。この多孔質石英ガラス体の表面に形成されるSiC、Si<sub>3</sub>N<sub>4</sub>等の膜（層）は、例えば図1に模式図として示すように、膜の断面組織構造が、実質的にセラミックス単身からなるガス不透過性の表層部分1と、セラミックスが多孔質石英ガラス体3の微細気孔内に浸透した浸潤組織構造の内層部分2とから成っていることが好ましい。

【0025】そしてまた、多孔質石英ガラス体をSiC被覆したセラミックス被覆石英ガラス体にあつては、前記SiC単身のガス不透過性表層部1の厚さは10乃至1000 $\mu$ mの範囲にあることが耐薬品性、ダスト発塵抑止性の点から好ましい。また、前記多孔質石英ガラス体の微細気孔内にSiCが浸透したSiC浸潤組織構造を有する内層部分2の厚さは3mm以上であることが強度、靱性を担保する観点や、石英ガラスとSiCとの熱膨張率差に基づく膜（層）剥離を回避する観点から好ましい。特に、強度の高い部材を必要とする場合は、SiCが部材成形体の中心部まで浸透した構造であることが好ましい。

【0026】上記SiC、Si<sub>3</sub>N<sub>4</sub>等のセラミックスを多孔質石英ガラス体の表面に被覆する方法としては、該多孔質石英ガラス体の表面に膜（層）を形成できる方法であれば特に限定されるものではないが、本発明における上記セラミックス被覆層形成に特に好適な方法として、高純度膜を形成でき、また上記内層部分を形成し易

いCVD成膜法(化学気相成長法)を挙げることができる。CVD成膜法は、堆積原料を気相(ガス)状態で供給し、化学反応を用いて基材表面に薄膜を形成する方法であり、反応を生じさせるエネルギーをどのような手段で与えるかにより熱CVD法、プラズマCVD法、光CVD法等がある。本発明では何れのCVD法も使用可能である。

【0027】一例として、熱CVD法を用いて多孔質石英ガラス体(厚さ20mm、直径100mmの円盤状基材)にSiC膜を被覆形成する場合についてその標準的10  
条件を述べると、通常炉内温度:1230℃、流入ガス:SiCl<sub>4</sub>(2.7SLM)、C<sub>3</sub>H<sub>8</sub>(0.9SLM)、H<sub>2</sub>(20SLM)、処理時間:5時間、の処理条件で、前記実質的にSiC単身から成る膜表層部分の厚さが100μm程度の被覆膜形成が可能である(SiC浸潤内層部分の厚さ3mm程度、それより内側の中心部にはシリカガラス質発泡体の部分が残る)。

【0028】このCVD法によるセラミックス膜(層)被覆処理工程において、膜(層)堆積処理を所定厚さになるまで複数回に分けて実施することがより好ましく、20  
例えば、上記処理時間を2.5時間ずつ2回に分け、その間に基材のフッ酸洗浄、乾燥操作を挟む等の処理手順を取ることが推奨される。例えば、支持具上に多孔質石英ガラス体をセットし、1回の化学気相成長によって所望の厚さ(例えば500~1000μm)のセラミックス層の表層部分を形成しようとする、多孔質石英ガラス体の支持具と接する部分にも、相応のセラミック膜が形成され多孔質石英ガラス体と支持具が接合された状態となり、多孔質石英ガラス体を炉出しする際に、このガラス体自身が破損してしまう不具合が生じてしまう。また、破損しないまでも前記支持具と接していた部分に十分にセラミックス膜(層)が形成されず、ガス不透過性が不十分になってしまう。前記したように、複数回に分けて実施することにより、炉内における基材載置治具の跡を残すことがなく、セラミックス被覆石英ガラス部材の表面全面に均一厚さの膜(層)を形成することができる。

【0029】本発明のセラミックス被覆石英ガラス体において、適当な多孔質石英ガラス体素材を準備し、これを所望の形状に加工して基材とする。次いでこの基材を、保持治具上に載置保持し、CVD炉内にセットする。そして、例えば上記条件下にCVD気相成長法により、セラミックス層を形成する。炉出し後、フッ酸洗浄等により被覆セラミックス層表面部分の汚染を取り除けば半導体製造プロセスにおける高純度雰囲気でのより好適に使用が可能となる。

【0030】このように形成されたSiC等のセラミックス被覆膜は、図1に示すように、表面から深さ10乃至1000μmまではSiC等のセラミックスのみから成り、それより深い部分、通常、表面から500μm以

上20mm以内、好ましくは3乃至5mm、の部分はSiC等のセラミックスが気孔(気泡)内部に浸透したセラミックス浸潤組織構造となる。通常、基材の中心部は多孔質石英ガラス体のまま残るが、中心部まで上記セラミックス浸潤構造とすることもでき、この部材は高強度で硬いものとなる。この基材の中心部に多孔質石英ガラス体のまま残存させる割合は、その用途により適宜設定することができるが、特に、半導体用の熱遮蔽材、断熱材としてより高い軽量性及び断熱性を確保するためにはセラミックス被覆石英ガラス体全体の50~80体積%とすることが好ましい。

【0031】上記セラミックス被覆石英ガラス体は、基材に多孔質石英ガラス体を用いているため、SiC等のセラミックス単独品より軽量で、断熱性能が高い。これらの性能をより高めるためには、多孔質石英ガラス体の例として上述したシリカ質多孔体及びシリカガラス質発泡体のうち、特に後者を用いることが好ましい。SiC等のセラミックス層(膜)を形成することにより、多孔質石英ガラス体の発塵を抑制することができる。特に、20  
表層が前記したようにCVD成膜されたセラミックス層の場合には、高純度であり、不純物放散抑制性も高い。また、表層がSiC等の耐食性セラミックス質のため耐薬品性が高く、部材全体を酸洗浄することが可能である。なお、板状から円状、更に複雑な形状まで、基材が加工可能である限り、任意の形状の部材を製作することができる等、利点も有する。

【0032】本発明のセラミックス被覆石英ガラス体にあつては、図2に示すように、任意の石英ガラス体4の基材表面に多孔質石英ガラス層3を設け、この表面をSiC等のセラミックス膜(層)を被覆したものであつてもよい。この態様のセラミックス被覆石英ガラス体は、30  
実質的に無気泡の透明または見掛密度2.0~2.19g/cm<sup>3</sup>の不透明の石英ガラス基材4の表面に多孔質石英ガラス層3を、例えば、融着等の手段により接合し、この多孔質石英ガラス層3の表面上記と同様にしてSiC等のセラミックス層(1、2)を形成することによって得られる。

【0033】多孔質石英ガラス層は、前記した多孔質石英ガラス体を融着等により接合したものであり、接合する多孔質石英ガラス層3の厚さは、部材の形状、用途等に応じて適宜設定されるが、通常の板状体の場合1乃至10mm程度の厚さに設定することが好ましい。前記多孔質石英ガラス層の厚さが薄い場合には、接合された多孔質石英ガラス層がセラミックス浸潤組織構造の内層部分2となり、多孔質石英ガラスのみの層がなくなる。そのため、硬く、高強度には成るが、靱性やフレキシビリティを若干欠く構造となる。

【0034】この態様のセラミックス被覆石英ガラス体は、多孔質石英ガラス層が緩衝層となり、石英ガラス体とSiC等のセラミックス被覆膜との間の熱膨張係数差

に起因する膜剥がれを防止でき、発塵を抑制できる。また、石英ガラス体と多孔質石英ガラス層との接合は、例えば融着等により容易に接合でき、両者の密着性も良好であるため、全体として丈夫な積層構造部材となる。また、セラミックス被覆石英ガラス体の表層がSiC等の高純度セラミックスで被覆されている場合には、不純物拡散抑止性に優れている。また、耐食性も高く、そのため部材全体を酸洗浄することもできる等数多くの利点も有する。この態様のセラミックス被覆石英ガラス体は断熱性の高い高強度構造材として有用性が高い。

#### 【0035】

【実施例1】「実施例1」シリカガラス質発泡体（見掛密度 $0.4\text{g/cm}^3$ 、気孔径 $200\sim 800\mu\text{m}$ 、平均線膨張係数 $5.6\times 10^{-7}/^\circ\text{C}$ （ $25\sim 900^\circ\text{C}$ ）、熱伝導率 $0.08\text{kcal/mhK}$ 、純度分析値（ppm）： $\text{Na}<0.2$ 、 $\text{K}<0.2$ 、 $\text{Li}<0.2$ 、 $\text{Mg}<0.2$ 、 $\text{Al}<1$ 、 $\text{Cu}<0.1$ ）を保温板形状（直径 $200\text{mm}\times$ 厚さ $3\text{mm}$ ）に加工し、この加工したシリカガラス質発泡体表面に、CVD炉中でSiC膜を $60\mu\text{m}$ （但し、実質的にSiC単身から成る表層部分の厚さ）形成した。なお、CVD膜成長処理条件は、炉内温度： $1230^\circ\text{C}$ 、流入ガス： $\text{SiCl}_4$ を $2.7\text{SLM}$ 、 $\text{C}_3\text{H}_8$ を $0.9\text{SLM}$ 、 $\text{H}_2$ を $20\text{SLM}$ 、処理時間 $2.5\text{hrs}$ とした。膜形成後炉出しし、洗浄・乾燥後そのまま保温板として使用した。この製品は、高い断熱効果を示すと共に常温と約 $800^\circ\text{C}$ 間における80回の繰り返し熱サイクル（使用期間2ヶ月、酸洗浄回数7回）使用においても表層からの発塵が、ほとんど見られなかった。

【0036】「実施例2」実施例1と同様のシリカ質多孔体（見掛密度： $1.85\text{g/cm}^3$ 、気孔分布 $20\sim 180\mu\text{m}$ ）を保温筒形状に加工し、この保温筒形状のシリカ質多孔体にCVD炉でSiC膜を $100\mu\text{m}$ （但し、実質的にSiC単身から成る表層部分の厚さ）を形成した。炉出しし、洗浄・乾燥後そのまま保温筒として使用した。表層の高純度SiC層が高純度雰囲気の使用も可能にすると共に雰囲気ガスによる腐食に耐え、2ヶ月以上の長期使用にもかかわらず表層からの発塵は、ほとんどなかった。また、該保温筒の使用中に表面にポリシリコン膜が付着し、 $\text{HF}+\text{HNO}_3$ 洗浄液で10回洗

【0037】「実施例3」炉壁用板状の見掛密度 $2.10\text{g/cm}^3$ の不透明石英ガラス体を用意し（縦 $100\text{mm}\times$ 横 $200\text{mm}\times$ 厚さ $3\text{mm}$ ）、この石英ガラス体の全表面に厚さ $10\text{mm}$ のシリカガラス質発泡体を融着した（発泡石英材性状：密度 $0.8\text{g/cm}^3$ 、気孔径 $50\sim 200\mu\text{m}$ 、平均線膨張係数 $5.6\times 10^{-7}/^\circ\text{C}$ （ $25\sim 900^\circ\text{C}$ ）、熱伝導率 $0.08\text{kcal/mhK}$ 、純度分析値（ppm）： $\text{Na}<0.2$ 、 $\text{K}<0.2$ 、 $\text{Li}<0.2$ 、 $\text{Mg}<0.2$ 、 $\text{Al}<1$ 、 $\text{Cu}<0.1$ ）。上記シリカガラス質発泡体を表面に接合した不透明石英ガラス体の該シリカガラス質発泡体層（多孔質石英ガラス層）の表面にCVD炉でSiC膜を $300\mu\text{m}$ の厚さに形成し、処理後炉出しし、洗浄・乾燥後そのまま使用した。製品は不透明石英ガラス基材、多孔質石英ガラス中間層、SiC表面層各層の層間密着性が良好で、長期間使用にも表層剥離等を生ずることがなかった。

10 【0038】「実施例4」反応管形状（外形 $308\text{mm}$ 、肉厚 $311\text{mm}$ 、長さ $350\text{mm}$ ）の透明石英ガラス体の全表面部に厚さ $3\text{mm}$ のシリカガラス質発泡体（実施例3と同じ性状のもの）を融着した。この反応管の該シリカガラス質発泡体（多孔質石英ガラス層）表面にCVD炉でSiC膜を $100\mu\text{m}$ 形成した。炉出しし、洗浄乾燥後そのまま使用した。表層の高純度SiC層が、高純度雰囲気下の使用も可能にすると共に雰囲気ガスによる腐食に耐え、2ヶ月以上の長期使用が可能であった。また、該反応管を使用中に、 $\text{HF}+\text{HNO}_3$ 洗浄液で10回洗浄したが、実質的にエッチングによる消耗がなく、変形等の不都合もなかった。

#### 【0039】

【発明の効果】本発明のセラミックス被覆石英ガラス体によれば、加熱冷却等の熱サイクルを長期間にわたって受けても、石英ガラスとセラミックス層（膜）との熱膨張率差に起因して生ずるセラミックス層（膜）の剥離や、亀裂の発生等を防止することができる。従って、本発明のセラミックス被覆石英ガラス体を、過酷な加熱・冷却熱サイクルを受ける環境下で長期間使用しても、劣化して部材表面から発塵することがなく、特に半導体製造プロセス用部材として好適に使用できる。

【0040】また、本発明のセラミックス被覆石英ガラス体の内、多孔質石英ガラス体にSiC等のセラミックス層を被覆した部材は、軽量で断熱性に優れているため保温板や熱遮蔽板、保温筒等の断熱部材として好適である。また、石英ガラス体に多孔質石英ガラス層を融着接合し、この多孔質石英ガラス層表面にセラミックス層を形成した態様の部材は、強靱性、耐食性及び耐摩耗性に優れ、炉壁材や反応管、ウエハ載置、熱処理用のサセプタ、ランプ加熱装置用あるいは面状ヒータ加熱装置用の各種部材等に好適に使用される。

#### 【図面の簡単な説明】

【図1】図1は、基材が多孔質石英ガラス体からなる態様の本発明のセラミックス被覆石英ガラス体の組織構造を模式的に示した断面図である。

【図2】図2は、石英ガラス体に多孔質石英ガラス層を融着した表面に、セラミックス層を形成した態様の本発明のセラミックス被覆石英ガラス体を模式的に示した図であって、（a）はその斜視図、（b）はその部分拡大断面図である。

(7)

特開2001-261375

1 1

1 2

【符号の説明】

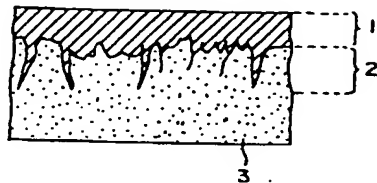
1 SiC被覆層(膜)(表層部分)

2 SiC被覆層(膜)(内層部分)

3 多孔質石英ガラス体(多孔質石英ガラス層)

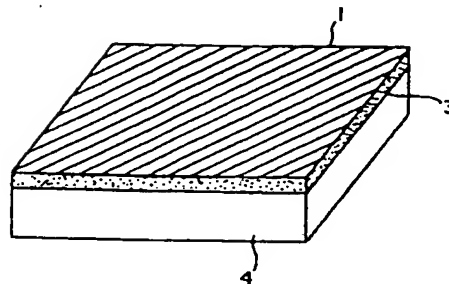
4 石英ガラス体

【図1】



【図2】

(a)



(b)



フロントページの続き

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Fターム(参考) 4G059 AA16 AB09 AC17 AC18 EA11  
EA12 EB01

4K030 AA03 AA17 BA37 CA06 FA10

JA10

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